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Studies on the β-ε Transformation in Cobalt-Nickel Alloys. II Microstructure of Transformation Relief*

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Synopsis

The dislocation model for the mechanism of diffusion-less transformation of the f.c.c. structure β into the c.p.h. ε in cobalt-nickel alloys was studied from electron-microscopic observation of relief markings on the crystal surface formed by the transformation. It can be considered that the transformation of β into ε proceeds through the motion of half-dislocations $(a/6)[1\bar{2}1]_\beta$, $(a/6)[\bar{2}11]_\beta$, and $(a/6)[11\bar{2}]_\beta$ in the (111) plane of a f.c.c. crystal. When these half-dislocations move in all directions over the crystal surface, tilts having three different inclinations to the surface are formed, which can be estimated to be 19°28′, −10°2′ and −10°2′, respectively, if the surface is parallel to the $(11\bar{2})_\beta$ plane, while under an external stress only one type of half-dislocations similar in direction to the stress can be considered to move, and then only one sort of the tilts is preferred. These surface tilts result in the relief markings on the crystal surface. The above results predicted theoretically were confirmed by electron-microscopic observation of markings, and it was found that the width of the zone of homogeneous displacement in marking (about 0.1∼1μ for 25 per cent nickel alloy) decreased slightly with decreasing nickel content, but in alloys containing less than a few per cent of nickel it decreased abruptly and in pure cobalt it was of the order of 100Å.

I. Introduction

The characteristic of the transformation of face-centred cubic lattice into close-packed hexagonal lattice lies in the simple relation of complete coincidence of the habit plane with the slip plane. The process of the transformation can be qualitatively interpreted by the motion of $(a/6)[11\bar{2}]_\beta$ (a denotes the lattice constant) of the half-dislocation in the slip plane of the face-centred lattice, as pointed out by Christian.[1]

In the present study, the details of the motion of atoms in the course of transformation were examined from the various properties attributable to the relief marking expected from the half-dislocation model by using an electron microscope.

II. Mechanism of propagation of transformation and genesis of markings

As to the crystallographical relation of face-centred cubic → close-packed hexagonal lattice transformation, Nishiyama[2] and Shoji[3] have confirmed the relation $(111)_\beta // (0001)_\varepsilon$ and $[11\bar{2}]_\beta // [1\bar{1}00]_\varepsilon$. That is, the transformation is accom-

* The 394th report of the Research Institute for Iron, Steel and Other Metals.
Reported in the Nippon Kinzoku Gakkai-Si, **19** (1955), 652.

(1) J. W. Christian, Proc. Roy. Soc., **A206** (1951), 51.
(2) Z. Nishiyama, Sci. Rep. Tohoku Univ., **25** (1936), 76.
(3) H. Shoji, Z. Krist., **84** (1932), 74.

plished by slipping of the pair of neighboring atomic planes in the $(111)_\beta$ plane of the face-centred cubic lattice in the direction of $[11\bar{2}]_\beta$ by the distance of $a/\sqrt{6}$. Fig. 1 shows the motion of atoms seen from the direction of $[\bar{1}10]_\beta$ during the tarnsformation. The atoms in the $(111)_\beta$ plane arranged in the order of ABC ABC and so on (open circles) in the face-centred lattice shift in the direction of $[11\bar{2}]_\beta$ (indicated by the arrows) by $a/\sqrt{6}$ and transform into the $(0001)_\varepsilon$ plane of close-packed hexagonal lattice (cloced circles) in the ABAB arrangement of atoms. During the tarnsformation, however, some dilatation and contraction, though very slight, are necessary, and in pure cobalt 0.16 per cent dilatation follows in two mutually perpendicular directions in the $(111)_\beta$ plane and 0.51 per cent contraction in the $[111]_\beta$ direction. The above shifting is nothing but a half-dislocation of $(a/6)$ $[11\bar{2}]_\beta$, and the propagation of the transformation in the direction of $[111]_\beta$ can be attributed to the same mechanism as that of the multiplication in twinning deformation proposed by Cottrell-Bilby.(4)* If the transformation progress under such a mechanism, macroscopic tilts will naturally appear on the surface of the specimen. For instance, taking the plane $(11\bar{2})_\beta$ represented by the dash line OP in the figure as the surface of the specimen, the half-dislocatoin $(a/6)$ $[11\bar{2}]_\beta$ in each of two atom planes would result in steps of $a/\sqrt{6}$ in each distance of $2a/\sqrt{3}$ and the mean of these steps would be the plane OP′ inclined by 19°28′ to the initial surface plane. On a given surface, the angle of inclination in the direction of the surface normal would be

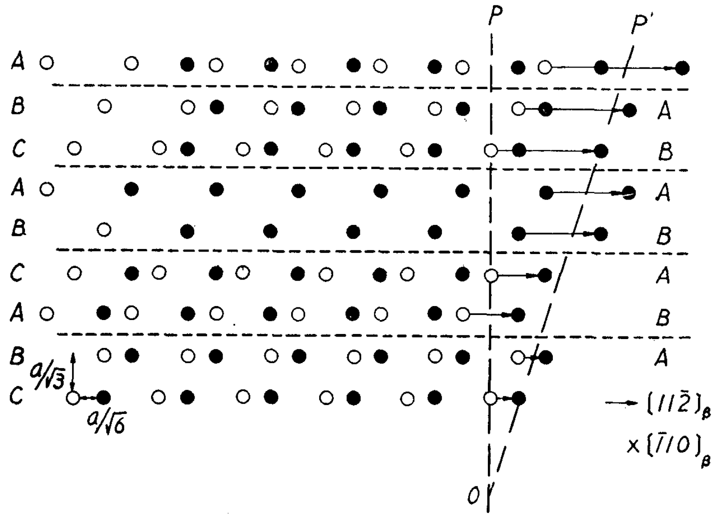

Fig. 1. Lattice relation of transformation from β (f.c.c) to ε (c.p.h).

$$\tan^{-1}\{A\cos\theta/(\operatorname{cosec}\chi + A\cos\theta\cot\chi)\},$$

and the angle of inclination in the parallel direction to (111) trace

$$\tan^{-1}\{A\sin\theta\cos\eta/(\operatorname{cosec}\chi + A\cos\theta\cot\chi),$$

here θ denotes the angle between surface normal and $[11\bar{2}]_\beta$, χ the angle between surface normal and $[111]_\beta$, η the angle between $(111)_\beta$ trace and the orthograph

(4) A. H. Cottrell and B. A. Bilby, Phil. Mag., **42** (1951), 573.

* J. W. Christian et al.(5) have proposed a mechanism in which the half dislocations $(a/6)$ $[11\bar{2}]_\beta$ turn around the pole dislocation $(2/3)a[111]_\beta$ with the Burgers vector perpendicular to the plane $(111)_\beta$ and climbing two planes each, and Suzuki(6) a more detailed mechanism taking the origin of this pole dislocation also into account.

(5) T. R. Anantharaman and J. W. Christian, Phil. Mag., **43** (1952), 1338.

(6) H. Suzuki, Conference of Japan Physical Society (Sendai, 1954).

of $[11\bar{2}]_\beta$ on the surface, and A tan 19°28′.

In the above, the reason of appearing of the relief markings on the surface of the spcimens is mentioned, but in actual structure the markings are not made of such single tilts as shown in Fig. 1, as reported.(7) The outstanding characteristics of the markings observed by an optical microscope may be stated as follows; (1) The cross section of the markings is of the ridges or valleys, but its tilts are not bilaterally symmetric and do not show regular decrease in the inclination as observed in transformed Ti. (2) The width of the markings is around $1\sim2\mu$ in Co–25 per cent Ni alloys, but tends to become narrow to some extent with the lowering of the Ni concentration. (3) In pure Co, no such marking is observable by optical microscope. (4) The markings extend precisely in parallel with the $(111)_\beta$ plane, and they are apt to from groups of markings in the same direction but the velocity of their growth is incomparably small to that of martensite of the "Umklapp" type in steel. The properties of the markings can be adequately explained by adopting the following mechanism for the propagation of the transformation.

As three kinds of the six half-dislocation forming the extended dislocation, $(a/2)[\bar{1}10]_\beta$, $(a/2)[0\bar{1}1]_\beta$ and $(a/2)[10\bar{1}]_\beta$, serve merely in eliminating the stacking faults of the close-packed hexagonal lattice, the half-dislocation fully taking part in the transformation consists only of three kind of the half-dislocations $(a/6)$ $[11\bar{2}]_\beta$, $(a/6)[1\bar{2}1]_\beta$ and $(a/6)[\bar{2}11]_\beta$.

Now, if the transformation should progress as shown in Fig. 1, a large strain would occur in the vicinity of P′, resulting in a shear tending to force back this region into the original position. In the case of Ti or Fe–Ni alloys*, such a large strain causes a plastic slip near P′ to eliminate the strain, but in the transformation under study, it causes only a transformation due to the other half-dislocations to elminate the strain, because the coincidence of the slip plane of the mother phase and the habit plane of the transformation despenses with such a slip. That is the transformation is accomplished by the three half-dislocations in the $(111)_\beta$ plane inclined by 120° to one another as shown in Fig. 2, and each half-dislocation is forced so as to negate the transformation strains caused by the other half-dislocations, resulting in the ridge-form structure of the cross-sections of the markings and their group formation in the same direction.

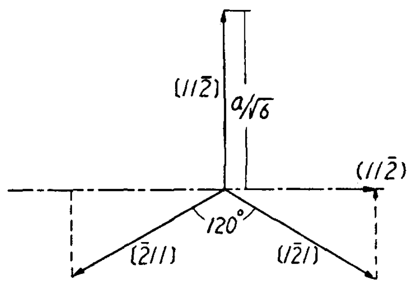

Fig. 2. Relation between three half dislocations.

If this picture of propagation mechanism proves true, as can be readily seen from Fig. 1, the markings formed on the surface of transformed specimens would consist of three types of inclined planes corresponding to those of the three half-dislocations,

(7) S. Takeuchi and T. Honma, Sci. Rep. RITU. **A9** (1957), 492.

* This type of transformation is called the "Schiebung" transformation and may be observed in Fe–Ni alloys containing less than 30 per cent nickel.

and the inclinations between these three planes should satisfy the simple mutual geometric relation as shown in Fig. 2. Considering the case of a crystal with surface in the $(11\bar{2})_\beta$ plane, as the case most convenient for verifying the assumption, the components of motion of the three half-dislocations in the direction perpendicular to the surface are $a/\sqrt{6}$, $-a/2\sqrt{6}$ and $-a/2\sqrt{6}$, respectively, and so the angles beween the tilts and the original surface planes would become 19°28′, −10°2′ and −10°2′, respectively, and in this case a cross-section structure is such as shown in Fig. 3 (a). On the other hand, the components of motion in the horizontal direction, that is, in the direction of $[\bar{1}10]_\beta$, would be 0, $-a/2\sqrt{2}$ a and $a/2\sqrt{2}$, respectively, and if a straight line is drawn perpendicular to the marking, this line would be retracted at the angles of 0°, −17°2′ and 17°2, respectively, as shown in Fig. 3 (b). If a replica of such a area is shadowed from the direction shown by the arrow and examined under an electronic microscope, such the structure as shown in Fig. 3(c) would be revealed, and the correctness of the above proposed propagation mechanism of the transformation may be seen from the surface markings.

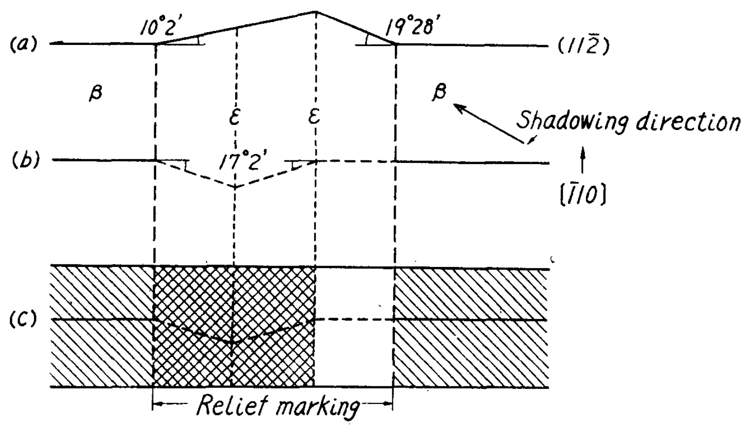

Fig. 3. Cross section of marking in $(11\bar{2})_\beta$ surface plane and corresponding electron microphotograph.
(a) Surface tilts on the $(11\bar{2})_\beta$ surface.
(b) Tilts in the $[\bar{1}10]_\beta$ orientation.
(c) Estimated electron microphotograph.

III. Materials and method of observation

Using electrolytic nickel and electrolytic cobalt, alloys with various Ni concentrations were prepared. These were melted in a high frequency electric furnace, forged into round rods, 5 mm in diameter, and cut into cylinders, each 1 cm in length. The cylindrical specimens were mechanically or electrolytically polished at room temperature, heated in molten alumina tubes in vacuum of the order of 10^{-4} mmHg at 1350°C for 3 hrs and then cooled in furnace.

Specimens of 30 per cent Ni which do not transform at room temperature were forced to transform under an external stress after the heat treatment and then electrolytically polished. The tested specimens were pure Co and Co–Ni alloys with Ni contents 2, 5, 10, 20, 25 and 30 per cent. On the surface of the specimens, grain boundaries, twin boundaries and stripe patterns formed by thermal etching were seen in which the relief markings were formed by the transformation. The surface structure was taken in replica by films of methyl-metaacryl shadowed with chromium and examined under an electron microscope.

The crystallographical orientation of the specimen surface was determined by measuring the angles between annealing twins or markings of four different directions in the same crystal grain.

IV. Results of observations

1. Cross-section of the marking

Photo. 1 shows the transformed structure of 25 per cent Ni alloy, an electron-microscopic photograph of a part showing numerous markings in one direction. The transformed part consists of a structure of different blackening demarcated by straight lines precisely to parallel the $(111)_\beta$ plane, the arrow in the photogaph showing the direction of shadowing. As a peculiarity of the electron microscopic structure of such markings, it may be pointed out that the degree of blackening in a band parallel to the same $(111)_\beta$ never exceed four levels. Because the blackening depends on the thickness of the film of chromium, that is, on the inclination to the shadowing direction, and there must be only four angles of inclination observable on a surface Now, as the part of intermediate tone increasing with the concentration of Ni represents the horizontal plane i. e., the untransformed area, it may be seen that there are only three kinds of surface tilts produced by transformation. As these three kinds of inclination are arranged so as to eliminate the strain due to the transformation in succession, as described in II above, the general appearance takes the form of ridges or valleys and is observed as markings under an optical microscope. Thus, it will be assured that the markings are composed of three tilts corresponding to half-dislocation in three different direction. Photo. 2 is the electron micrograph of parallel markings in a Co–10 per cent Ni alloy, in which the same relation of the tilts as the above-mentioned can be observed. Many fine patterns in the picture are due to thermal etching.

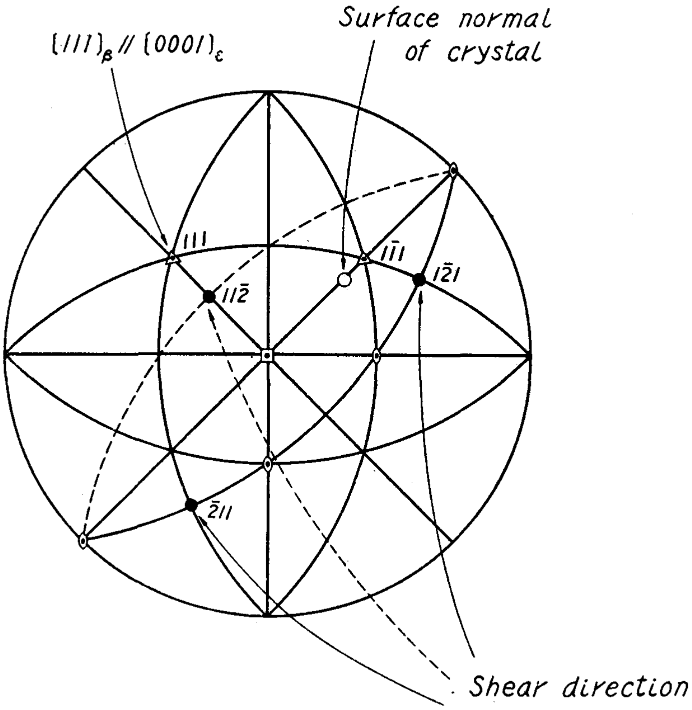

Fig. 4. Orientation of the observed crystal surface and shear directions.

2. Measurement of displacement of atoms

In Photo. 3 is shown an electron-microscopic picture of markings in a surface of the crystal having the orientation shown in Fig. 4. The uniform part of intermediate blackness is an untransformed, the many straight parallel stripes representing a pattern produced by thermal etching*. The broad zone structure

* In most cases appearing along the $\{100\}_\beta$ plane of the face-centred cubic lattice.

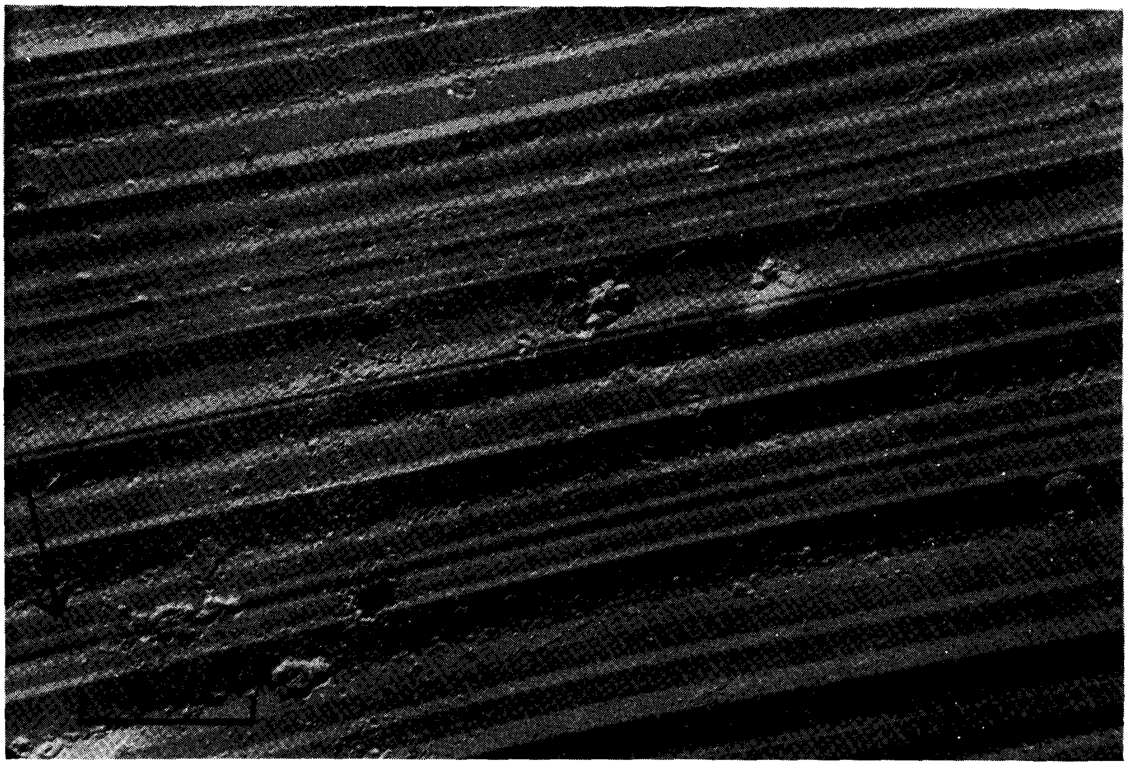

Photo 1. Electron microphotograph of markings in a 25% Ni alloy. the arrow represents shadowing direction. (methyl-metaacryl-Cr method)

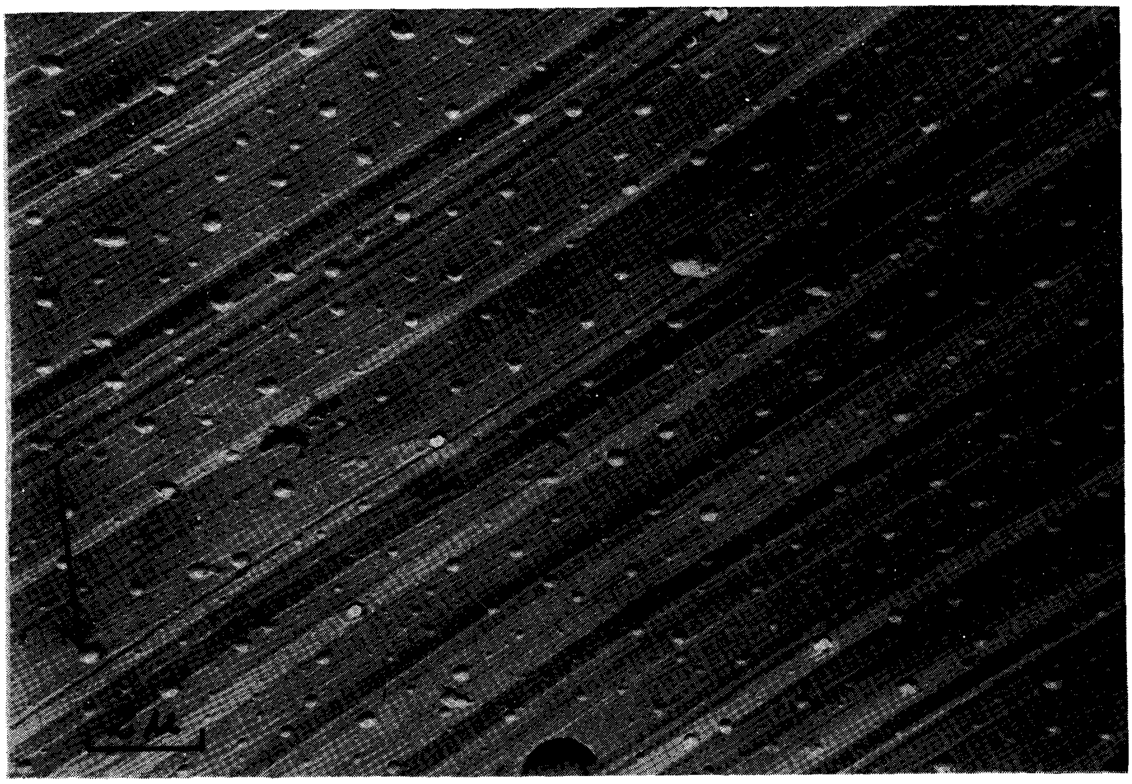

Photo 2. Electron microphotograph of markings in a 10% Ni alloy. (fine striations are due to thermal etching)

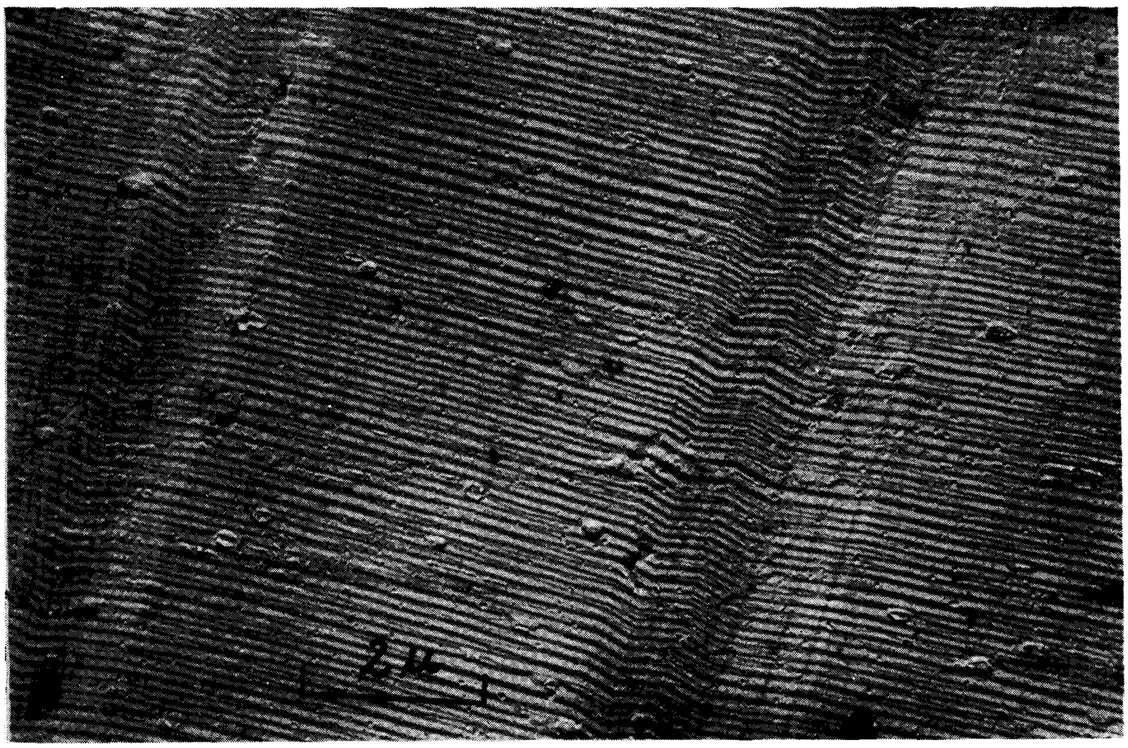

Photo 3. Displacement of striations of thermal etching by the markings on the nearly $\{112\}_\beta$ surface. (Co-25% Ni alloy)

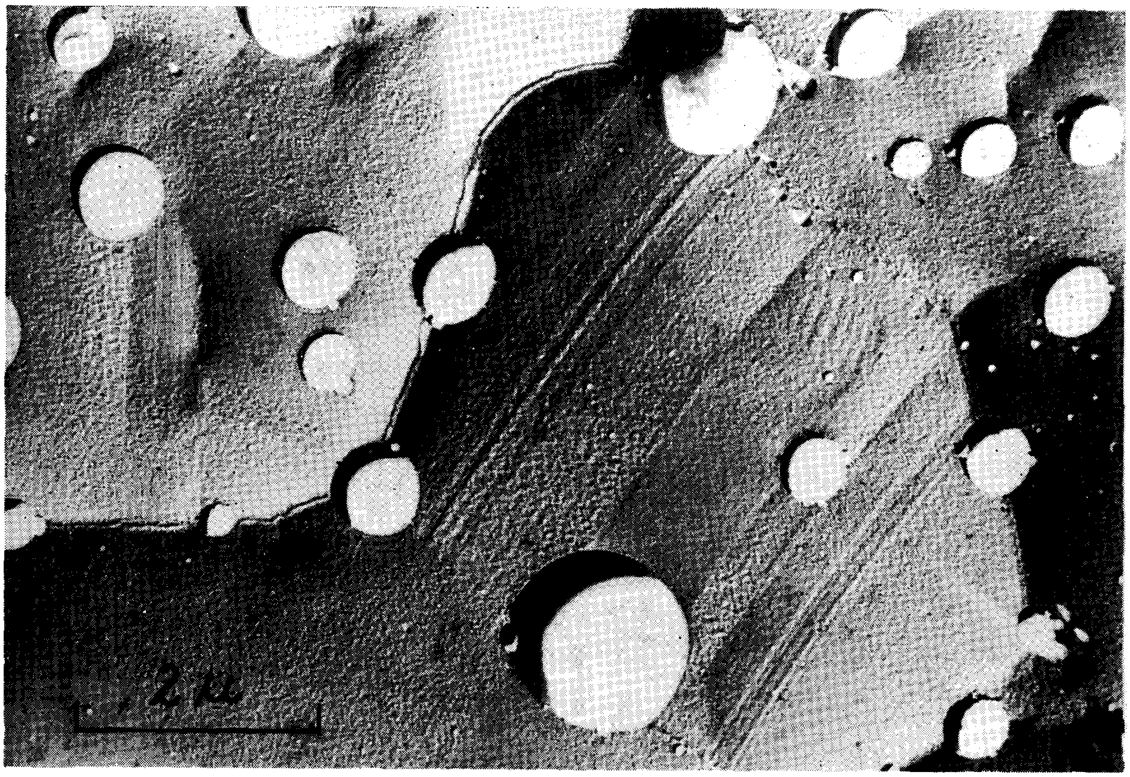

Photo 4. Electron microphotograph of markings in pure cobalt.

running vertically on the left-hand side is a marking due to transformation. Comparing this structure with the shema in Fig. 3, it can be seen that the surface inclination, i, e,, the relative blackness and the way in which the straight striae produced by thermal etching are crooked at the tilts are in agreement with what have been predicted above. If it is taken into account that the thermal etching striae incline to this $(111)_\beta$ trace by 6°, and that the orientation of the surface is inclined to the poles of $(111)_\beta$, $(11\bar{2})_\beta$ as shown in Fig. 4, and then the angles by which the stripes are cooked, are computed, it will be obtained $-3°12'$. $-14°18'$ and $18°6'$, respectively, that is, these values are in agreement with the values $-4°$, $-14°30'$ and 18° measured from Photo. 3. Thus, it can be verified that the mechanism of propagation of the transformation depends on the motions of the three kinds of half-dislocation of $a/6[11\bar{2}]_\beta$ type inclined by 120° to one another in the $(111)_\beta$ plane.

3. Width of the homogeneous tilted zone and the Ni concentration

As seen in Photo. 1, the width of the tilted zone forming the markings is of the order of $0.1\sim1\mu$. This width is of importance as it shows how many atomic planes are climbed by the half-dislocation in the direction of the $[111]_\beta$ as shown in Fig. 1. In the specimen of 25 per cent Ni alloy, the half-dislocation should have climbed up in helicoid motion around the pole dislocation from 250 to 2,500 times by the distance of $2\,a/\sqrt{3}$ in each climbing. This quantity of multiplication will be determined by the difficultness of the motion of the succeeding half-dislocation which eliminates the strain occurred by the transformation due to the preceding half-dislocation, that is, by the critical shear stress of the half-dislocation. So, it can be considerd that the width of a homogeneous tilted zone depends on the temperature and the concentration of the solute atoms. Fig. 5 shows the mean width of zone of many observed markings versus the Ni concentration. It will be seen that the width suddenly drops at about 5 per cent Ni content. Photo. 4 is the appearance under an electronic microscope of a pure Co crystal, which shows no marking under an optical microscope. Extremely narrow markings, about $100\sim300$ Å in width, are arranged paraplle1 to $\{111\}_\beta$ in a large number. Photo. 5 shows such narrow markings in Co-2 per cent Ni alloy. These confirm that the transformation in pure cobalt is also propagated by the same mechanism as in Co-Ni alloys mentioned above, only the quantity of multiplication of the half-dislocations being of the order one decimal lower than that in Co-Ni alloys. This marked narrowing down of the zones might be induced mainly by the lowering of the critical shear stress of the half-dislocations caused by the weakening of the Cottrell's locking;

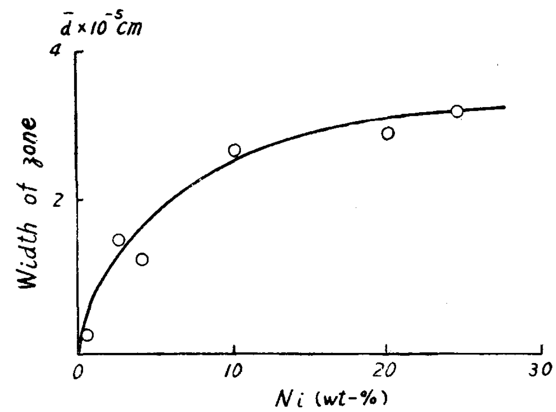

Fig. 5. Relation between width of zone of homogeneous displacement in surface tilt and Ni content.

force(8) due to the reduction of the Ni concentration.

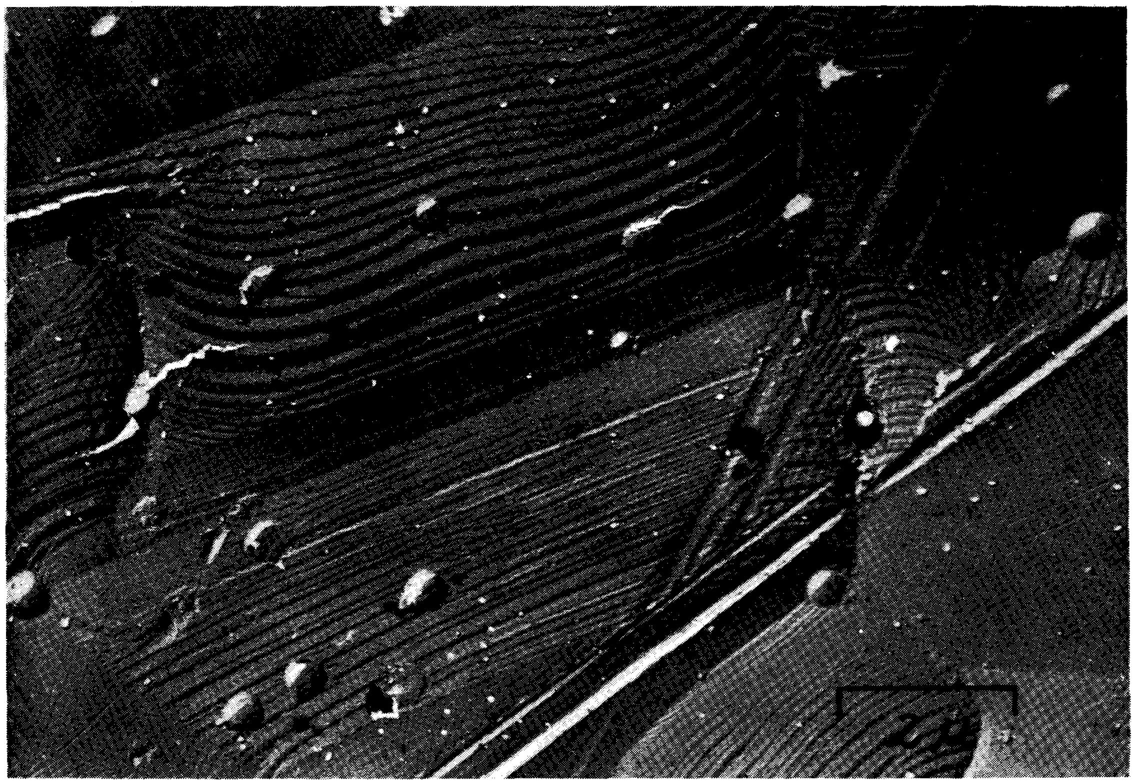

Photo 5. Electron microphotograph of markings and thermal etching figure. (Co-2%Ni alloy)

4. Transformation by external stress

The foregoing explanations are all concerned with spontaneous transformation, i. e. the transformation due to cooling, but it may be predicted that the structure will be very different when the transformation is occurred by an external stress as reported previously(7). In such a case, of the three kinds of half-dislocations partaking in the transformation, the one most effective in negating the strain due to the external force will come into preference, and so the marking is formed by the tilt corresponding to the motion of such preferred half-dislocation alone, and has a cross-section structure as illustrated in Fig. 1. Photo. 6 is an electron-microscopic photograph of the surface of a compressed specimen of 30 per cent Ni alloy that does not undergo spontaneous transformation at room temperature. The markings are formed by tilts of one direction alone which closely resemble the slip bands in plastic deformation.

5. Microstructure of the markings at twin boundaries and grain boundaries

As reported in the previous paper, many areas in which markings pass through twin boundaries or grain boundaries continuously have been observed under an optical microscope According to electron microscopic observations of such area, it was seen that individial tilted planes which compose a marking do not continue at such boundaries. Photos. 7 and 8 are the electron microphotographs of the

(8) A. H. Cottrell, Rep. Bristol. Conference, (1948. London), 30.

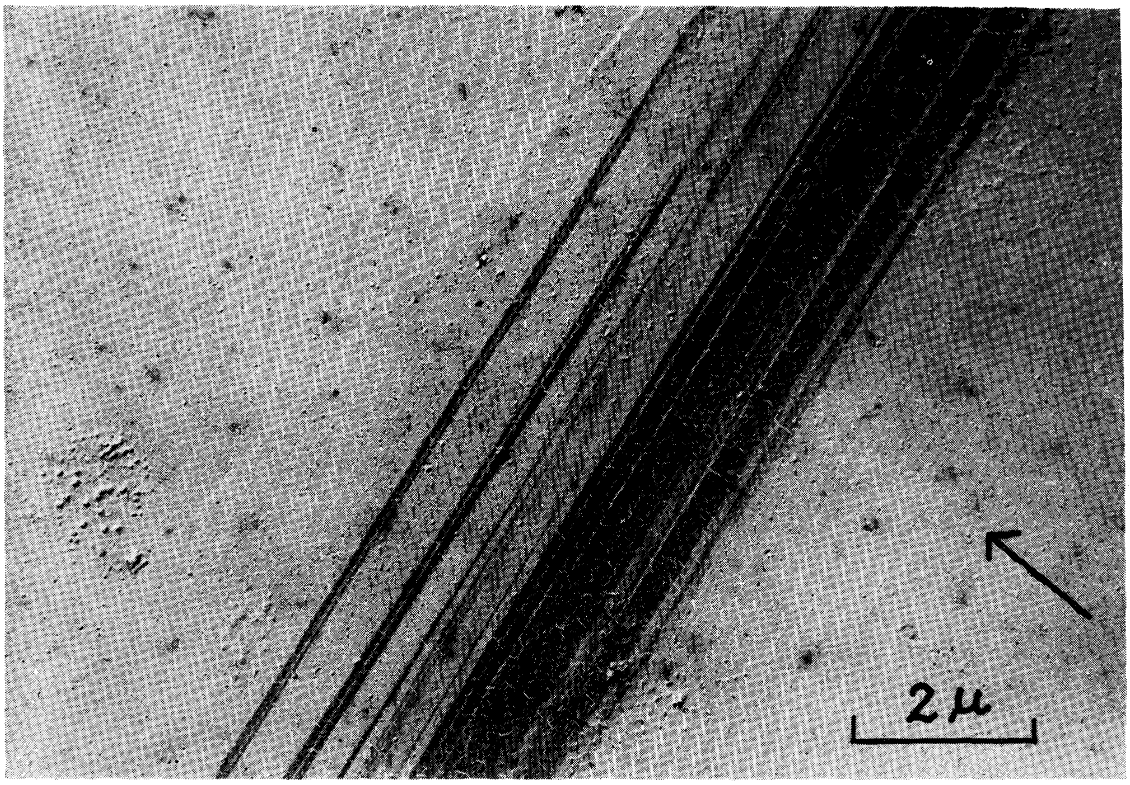

Photo 6. Electron microphotograph of markings due to applied stress in Co-30% Ni alloy.

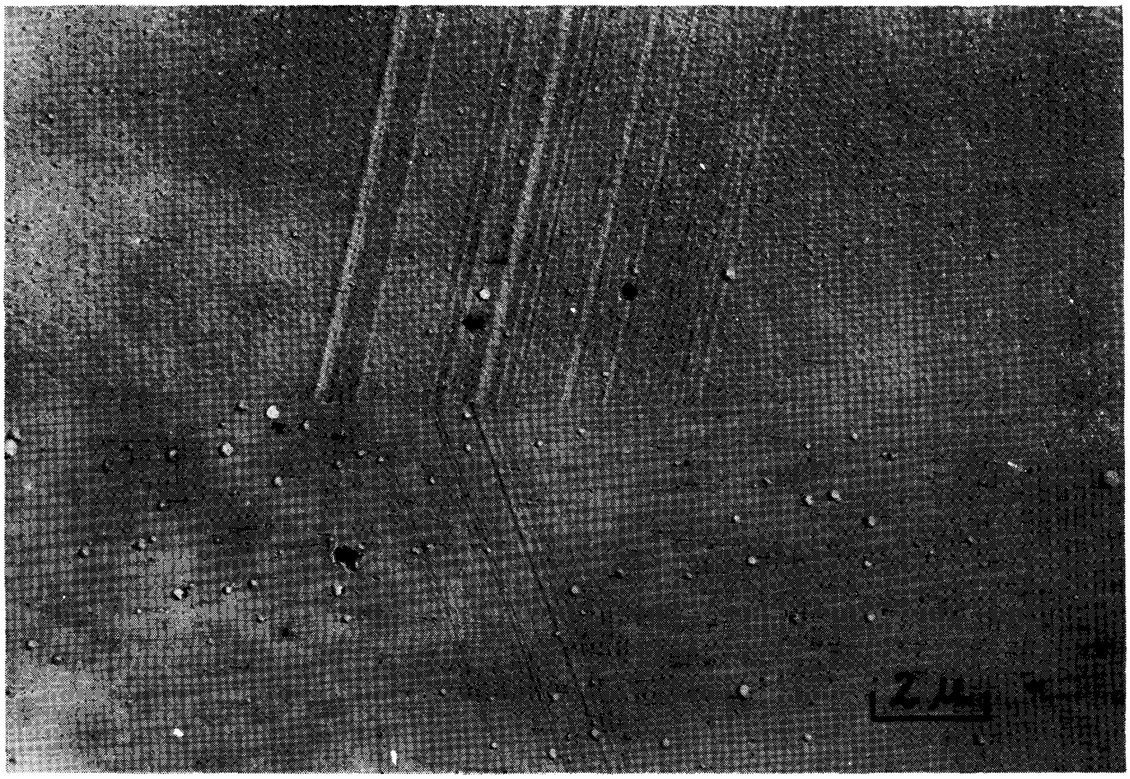

Photo 7. Electron microphotograph of markings at twin boundary. (Co-20%Ni alloy)

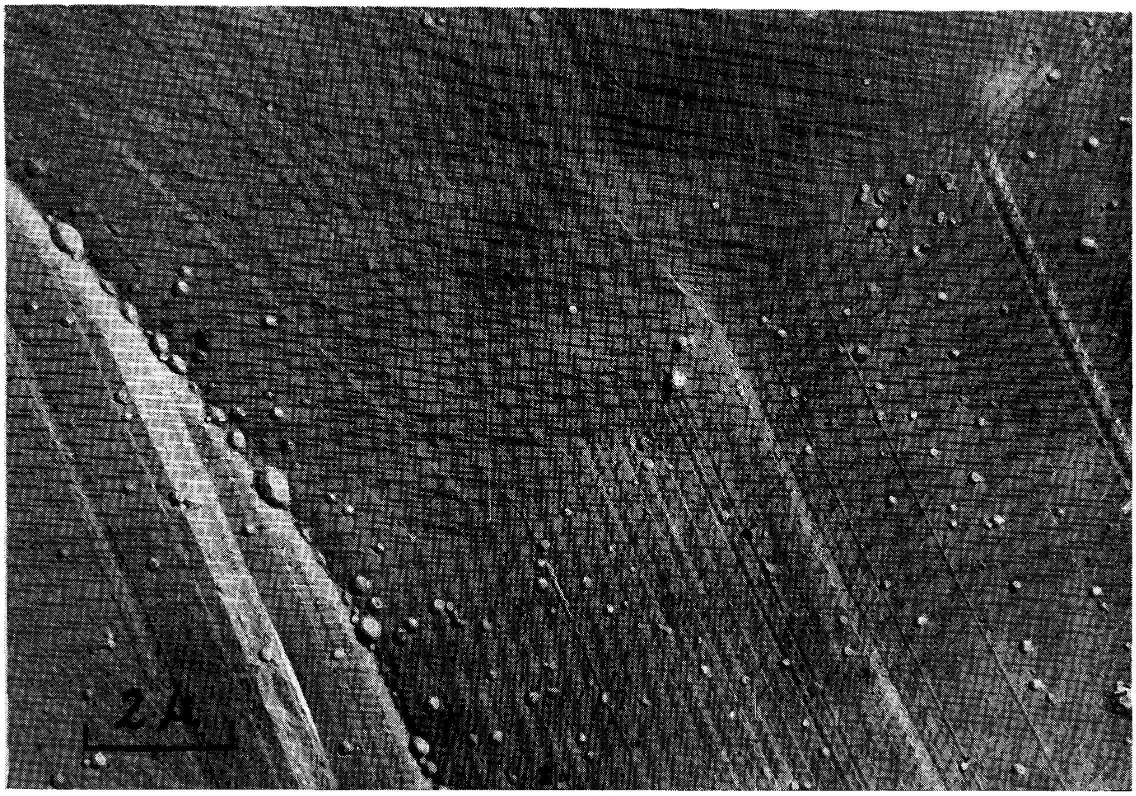

Photo 8. Electron microphotograph of markings at twin boundary. (Co-10%Ni alloy)

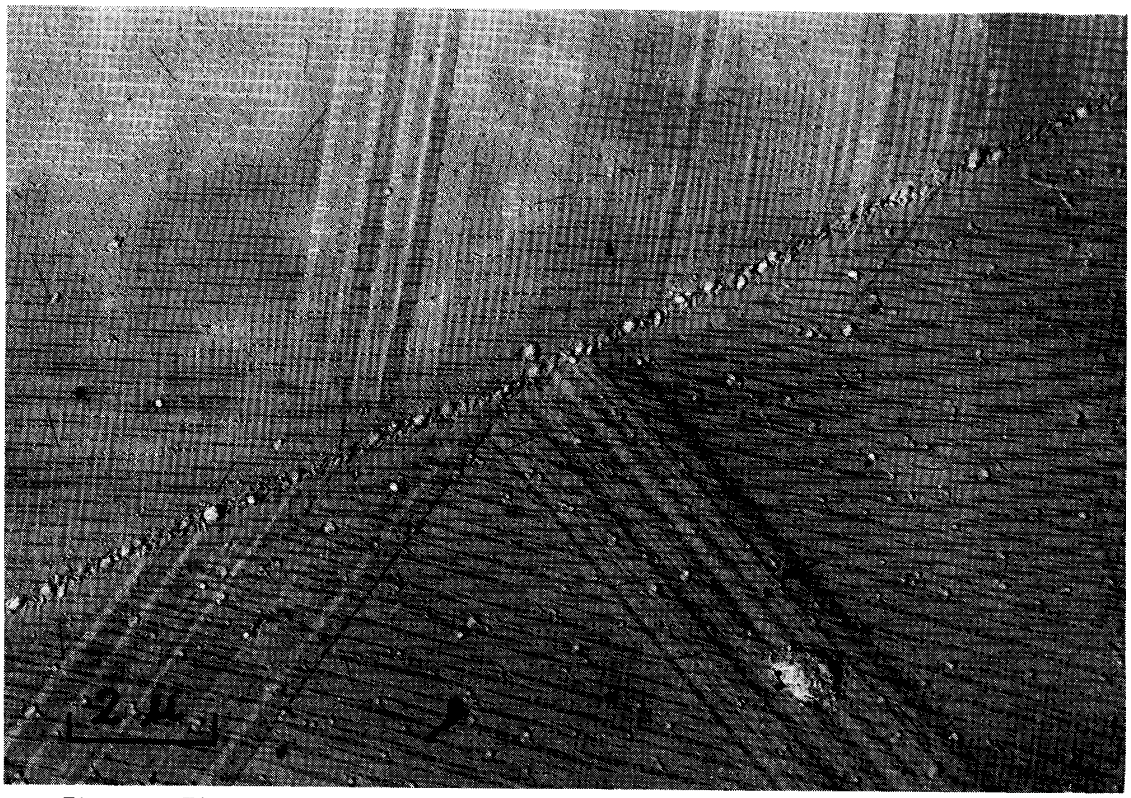

Photo 9. Electron microphotograph of markings at grain boundary. (Co-10%Ni alloy)

markings at the twin boundary. Especially, such markings as shown in Photo. 8 are these considered as if they were continuous at the twin boundary under the optical microscopic observation. Photo. 9 shows the markings at the grain boundary, in which no tilted plane passing through the grain boundary is observable.

Summary

The mechanism of propagation of the face-centred cubic → close-packed hexagonal lattice transformation in Co-Ni alloy was studied from the observations of the microstructure of the relief-markings with electronic microscope. The results may be summarized as follows:

(1) Each marking consists of three types of tilts brought about by the motion of three kinds of $(a/6)[11\bar{2}]_\beta$ type half-dislocations in the $(111)_\beta$ plane.

(2) The marking in specimens transformed by external force consists of tilts of one direction alone as in plastic slips, and so the form of the markings depends on the way of eliminating the strain producd by the transformation.

(3) The width of a tilt, that is, the distance by which the half-dislocation is multiplied in the direction of $[111]_\beta$ is of the order of $0.1\sim1\mu$ in $10\sim25$ per cent Ni alloys and decreases abruptly when the Ni content is reduced below ca. 5 per cent, becoming $100\sim300$Å in pure Co.

Acknowledgments

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